This Page Is Inserted by IFW Operations and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problem Mailbox.

MAGNESIUM-BASED CASTING ALLOYS HAVING IMPROVED ELEVATED TEMPERATURE PERFORMANCE

Patent Number:

WO0144529

Publication date:

2001-06-21

Inventor(s):

LABELLE PIERRE (CA); PEKGULERYUZ MIHRIBAN O (CA)

Applicant(s)::

NORANDA INC (CA); LABELLE PIERRE (CA); PEKGULERYUZ MIHRIBAN O

(CA)

Requested Patent:

WO0144529

Application

Number:

WO2000CA01527 20001214

Priority Number(s):

US19990461538 19991215

IPC Classification:

C22C23/02

EC Classification:

Equivalents:

Abstract

A magnesium-based casting alloy having good salt-spray corrosion resistance and improved creep resistance, tensile yield strength and bolt-load retention, particularly at elevated temperatures of at least 150 DEG C, is provided. The inventive alloy comprises, in weight percent, 2 to 9% aluminum and 0.5 to 7% strontium, with the balance being magnesium except for impurities commonly found in magnesium alloys.

Data supplied from the esp@cenet database - 12

-- &DND-WOOLAA529&PN=WOOL44529&CURDRAW=0&DB=119/07/01

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date 21 June 2001 (21.06.2001)

PCT

(10) International Publication Number WO 01/44529 A1

(51) International Patent Classification7:

C22C 23/02

- PCT/CA00/01527 (21) International Application Number:
- (22) International Filing Date: 14 December 2000 (14.12.2000)
- (25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data: 09/461,538

15 December 1999 (15.12.1999)

(71) Applicant (for all designated States except US): NO-RANDA INC. [CA/CA]; BCE Place, 181 Bay Street, Suite 4100, P.O. Box 755, Toronto, Ontario M5J 2T3 (CA).

(72) Inventors; and

(75) Inventors/Applicants (for US only): PEKGULERYUZ, Mihriban, O. [CA/CA]; 213-165 Frobisher, Pointe-Claire, Quebec H9R 4R8 (CA). LABELLE, Pierre [CA/CA]; 137 Chemin du Lac Maillé, St-Hippolyte, Quebec J8A 1J8 (CA).

- (74) Agents: DUBUC, Jean, H. et al.; Goudreau Gage Dubuc, The Stock Exchange Tower, 800 Place Victoria, Suite 3400, P.O. Box 242, Montreal, Quebec H4Z 1E9 (CA).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

- With international search report.
- Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: MAGNESIUM-BASED CASTING ALLOYS HAVING IMPROVED ELEVATED TEMPERATURE PERFORMANCE

(57) Abstract: A magnesium-based casting alloy having good salt-spray corrosion resistance and improved creep resistance, tensile yield strength and bolt-load retention, particularly at elevated temperatures of at least 150 °C, is provided. The inventive alloy comprises, in weight percent, 2 to 9% aluminum and 0.5 to 7% strontium, with the balance being magnesium except for impurities commonly found in magnesium alloys.

MAGNESIUM-BASED CASTING ALLOYS HAVING IMPROVED ELEVATED TEMPERATURE PERFORMANCE

FIELD OF THE INVENTION

5

10

15

The present invention generally relates to magnesium based casting alloys having improved elevated temperature performance and more particularly relates to magnesium-aluminum-strontium alloys having good salt-spray corrosion resistance and good creep resistance, tensile yield strength and bolt-load retention, particularly at elevated temperatures of at least 150°C.

BACKGROUND OF THE INVENTION

Magnesium-based alloys have been widely used as cast parts in the aerospace and automotive industries and are mainly based on the following four systems:

Mg-Al system (i.e., AM20, AM50, AM60); Mg-Al-Zn system (i.e., AZ91D); Mg-Al-Si system (i.e., AS21, AS41); and Mg-Al-Rare Earth system (i.e., AE41, AE42).

20

25

Magnesium-based alloy cast parts can be produced by conventional casting methods which include diecasting, sand casting, permanent and semi-permanent mold casting, plaster-mold casting and investment casting.

These materials demonstrate a number of particularly advantageous properties that have prompted an increased demand for magnesium-based alloy cast parts in the automotive industry. These properties include low density, high strength-to-weight ratio, good castability, easy machineability and good damping characteristics.

10

15

· 20 ·

25

AM and AZ alloys, however, are limited to low-temperature applications where they are known to lose their creep resistance at temperatures above 140°C. AS and AE alloys, while developed for higher temperature applications, offer only a small improvement in creep resistance and/or are expensive.

It is therefore an object of the present invention to provide relatively low cost magnesium-based alloys with improved elevatedtemperature performance.

It is a more particular object to provide relatively low cost magnesium-aluminum-strontium alloys with good creep resistance, tensile yield strength and bolt-load retention, particularly at elevated temperatures of at least 150°C, and good salt-spray corrosion resistance.

SUMMARY OF THE INVENTION

The present invention therefore provides a magnesium-based casting alloy comprising, in weight percent, 2 to 9% aluminum and 0.5 to 7% strontium with the balance being magnesium except for impurities commonly found in magnesium alloys.

The foregoing and other features and advantages of the present invention will become more apparent from the following description and accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Particular features of the disclosed invention are illustrated by reference to the accompanying drawings in which:

FIG. 1 is a photomicrograph showing the microstructure of a diecast alloy of the present invention, hereinafter referred to as alloy A1;

10

15

20

25

FIG. 2 is a photomicrograph showing the microstructure of another diecast alloy of the present invention, hereinafter referred to as alloy A2;

FIG. 3 is a photomicrograph showing the microstructure of permanent mold cast alloy AD9; and

FIG. 4 is a photomicrograph showing the microstructure of permanent mold cast alloy AD10.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The magnesium-based casting alloys of the present invention are relatively low cost alloys that demonstrate improved creep resistance, tensile yield strength and bolt-load retention at 150°C. The inventive alloys also demonstrate good salt-spray corrosion resistance.

As a result of the above-identified properties, the inventive alloys are suitable for use in a wide variety of applications including various elevated temperature automotive applications such as automotive engine components and housings for automotive automatic transmissions.

The inventive alloys generally will have a preferred average % creep deformation at 150°C of \leq 0.06% for diecast alloys and \leq 0.03% for permanent-mold cast alloys. In addition, the alloys generally will have an average bolt-load-loss (measured as additional angle to re-torque) at 150°C of \leq 6.3° for alloys in the diecast state and \leq 3.75° for alloys in the permanent-mold cast state.

In regard to tensile properties, the inventive alloys will generally have an average tensile yield strength (ASTM E8-99 and E21-92 at 150°C) of >100 megapascals (MPa) for diecast alloys and >57MPa for permanent-mold cast alloys.

The average resistance of the inventive alloys to salt-spray corrosion, when measured in accordance with ASTM B117, is preferably ≤0.155 milligrams per square centimeter per day (mg/cm²/day) for alloys in the diecast state.

5

In general, the magnesium-based alloys of the present invention are 100% crystalline alloys that contain, in weight percent, 2 to 9% aluminum and 0.5 to 7% strontium, with the balance being magnesium. Main impurities commonly found in magnesium alloys, namely - iron (Fe), copper (Cu) and nickel (Ni), are preferably kept below the following amounts(by weight): Fe \leq 0.004%; Cu \leq 0.03%; and Ni \leq 0.001% to ensure good salt-spray corrosion resistance.

In addition to the above components, the alloys of the present invention may contain the elements manganese (Mn) and/or zinc (Zn) in the following proportions (by weight): 0 - 0.60% Mn; and 0 - 0.35% Zn.

15

20

10

In a preferred embodiment, the inventive magnesium-based alloys contain, in weight percent, 4 to 6% aluminum, 1 to 5% strontium (more preferably 1 to 3%), 0.25 to 0.35% manganese and 0 to 0.1% zinc, with the balance magnesium. In yet a more preferred embodiment, the inventive alloys contain, in weight percent, 4.5 to 5.5% aluminum, 1.2 to 2.2% strontium, 0.28 to 0.35% manganese and 0 to 0.05% zinc, with the balance magnesium.

The inventive alloys may advantageously contain other additives provided any such additives do not adversely impact upon the elevated temperature performance and salt-spray corrosion resistance of the inventive alloys.

25

The inventive alloy can be produced by conventional casting methods which include diecasting, permanent and semi-permanent mold

20

casting, sand-casting, squeeze casting and semi-solid casting and forming. It is noted that such methods involve solidification rates of $< 10^2$ K/sec.

In a preferred embodiment, the alloy of the present invention is prepared by melting a magnesium alloy (e.g., AM50), stabilizing the temperature of the melt between 675 and 700°C, adding a strontium aluminum master alloy (e.g., 90-10 Sr-Al master alloy) to the melt and then casting the melt into a die cavity using either diecasting or permanent mold casting techniques.

The microstructure of the alloys obtained is described as follows. The matrix is made up of grains of magnesium having a mean particle size of from about 10 to about 200 micrometers (µm) (preferably from about 10 to about 30µm for alloys in the diecast state and greater than 30µm for alloys in the permanent mold cast state). The matrix is reinforced by precipitates of intermetallic compounds dispersed homogeneously therein, preferably at the grain boundaries, that have a mean particle size of from about 2 to about 100µm (preferably from about 5 to about 60µm for diecast alloys and slightly larger for permanent mold cast alloys).

Scanning electron microscopy of the inventive alloys show that the diecast alloys contain Al-Sr-Mg containing second phases approximately 2 to 30μ m long and approximately 1 to 3μ m thick while the permanent mold cast alloys contain Al-Sr-Mg containing second phases approximately 10 to 30μ m long and approximately 2 to 10μ m thick

As best shown by the scanning electron micrographs of FIGS. 1 and 2, the microstructures of inventive diecast alloys A1 and A2, which have a chemical composition as described in Table 1 hereinbelow, contain Al-Sr-Mg containing second phases approximately 25 μ m long and 2μ m thick.

As best shown by the scanning electron micrographs of FIGS. 3 and 4, the microstructures of inventive permanent mold cast alloys AD9 and AD10, which have a chemical composition as described in Table 1 hereinbelow, contain Al-Sr-Mg containing second phases approximately 30µm long and 5µm thick.

The present invention is described in more detail with reference to the following Examples which are for purposes of illustration only and are not to be understood as indicating or implying any limitation on the broad invention described herein.

10

15

25

WORKING EXAMPLES

Components Used

AM50 - a magnesium alloy containing 4.17% by weight of aluminum and 0.32% by weight of manganese obtained from Norsk-Hydro, Bécancour, Québec, Canada.

90-10 Sr-Al - a strontium-aluminum master alloy containing 90% by weight

master alloy strontium and 10% by weight aluminum obtained from Timminco Metals, a division of Timminco Ltd., Haley, Ontario, Canada.

AZ91D - a magnesium alloy containing 8.9 (8.3-9.7)% by weight aluminum, 0.7 (0.35-1.0)% by weight zinc and 0.18 (0.15-0.5)% by weight manganese obtained from Norsk-Hydro.

AM50 - a magnesium alloy containing 4.7 (4.4-5.5)% by weight aluminum and 0.34 (0.26-0.60)% by weight manganese obtained from Norsk-Hydro.

	AS41 -	a magnesium alloy containing 4.2-4.8 (3.5-5.0)% by
		weight aluminum and 0.21 (0.1-0.7)% by weight
		manganese obtained from
		The Dow Chemical Company, Midland, MI.
5	AM60B -	a magnesium alloy containing 5.7 (5.5-6.5)% by
		weight aluminum and 0.24 (0.24-0.60)% by weight
		manganese obtained from Norsk-Hydro.
	AE42 -	a magnesium alloy containing 3.95 (3.4-4.6)% by
		weight aluminum and 2.2 (2.0-3.0)% by weight of rare
10		earth elements and a minimum of 0.1% by weight
	• • •	manganese obtained from Magnesium Elektron, Inc.,
•		Flemington, NJ.
	A380 -	an aluminum alloy containing 7.9% by weight silicon
		and 2.1% by weight zinc obtained from Roth Bros.
15		Smelting Corp., East Syracuse, NY.
	•	

Sample Preparation Alloys A1 and A2

Two different alloys were prepared by: charging ingots of AM50 into an 800 kilogram (kg) crucible positioned in a Dynarad MS-600 electric resistance furnace; melting the charge; stabilizing the temperature of the melt at 670°C; and adding 90-10 Sr-Al master alloy to the melt.

The temperature of the melt was maintained at 670°C for 30 minutes, stirred and then chemical analysis samples taken by pouring equal quantities of the melt into copper spectrometer molds.

The chemical analysis samples were analyzed using ICP mass spectrometry. The chemical composition of the prepared alloys, namely -

10

15

A1 and A2, are shown in Table 1 hereinbelow. The recovery rate of strontium was determined to be approximately 90%.

The temperature of the melt was cooled to 500°C while the ICP chemical analysis was carried out on the melt samples. The melt temperature was monitored by both a furnace controller and by a hand-held K-type thermocouple connected to a Fluke-51 digital thermometer.

During melting and holding, the melt was protected under a gas mixture of 0.5% SF $_6$ - 25% CO $_2$, balance air.

The molten metal was die-cast using a 600-tonne Prince (Prince-629) cold-chamber diecasting machine to produce diecast flat-tensile specimens measuring $8.3 \times 2.5 \times 0.3$ cm (gage 1.5×0.6 cm), round tensile specimens measuring 10×1.3 cm (gage 2.54×0.6 cm), cylindrical test specimens measuring 4×2.5 cm and corrosion test plates measuring $10 \times 15 \times 0.5$ cm.

Operating parameters used for the cold-chamber diecasting machine are shown below.

Operating Parameters	AZ91D	AS41	AE42	AM60	A380	A1	A2
Alloy Temp. (°C)	680	720	750	750	750	720	720
Temperature Of Metal Before Injection (°C)	250	300	300	300	300	275	275
Pressure (MPa)	13.8	13.8	13.8	13.8	13.8	13.8	13.8
Piston length (cm)	3.8/29.2	3.8/29.2	3.8/29.2	3.8/29.2	3.8/29.2	3.8/29.2	3.8/29.2
Base speed (cm/sec)	28-51	28-51	28-51	28-48	28-48	28-51	28-51
Fast speed (cm/sec)	384-516	315-498	368-587	417	312-330	384-516	384-516
Average cycle time (sec)	44-58	43-73	46-50	43	42-49	44-58	44-58

15

Operating Parameters	AZ91D	AS41	AE42	AM60	A380	A 1	A2
Average die opening time (sec)	30-44	29-54	32-36	18-29	18-35	30-44	30-44
Die Lubricant	Rdi-3188	Rdl-3188	Rdl-3188	Rdl-3188	Rdl-3188	Rdl-3188	Rdl-3188

Alloys AD9-AD14

Six different alloys were prepared by: charging 250g ingots of AM50 into a 2 kg steel crucible positioned in a Lindberg Blue-M electric resistance furnace; melting the charge; stabilizing the temperature of the melt between 675 and 700°C; and adding small pieces of 90-10 Sr-Al master alloy to the melt.

The temperature of the melt was maintained at either 675°C for 30 minutes or at 700°C for 10 minutes, stirred and then chemical analysis samples taken by pouring equal quantities of the melt into copper spectrometer molds.

The chemical analysis samples were analyzed using ICP mass spectrometry. The chemical composition of the prepared alloys, namely - AD9 to AD14, are shown in Table 1 hereinbelow. The recovery rate of strontium was determined to be 87-92%.

The temperature of the melt was measured by a K-type Chromel-Alumel thermocouple immersed in the melt.

During melting and holding, the melt was protected under a gas mixture of 0.5% SF₆, balance CO₂.

The molten metal was permanent mold cast using copper permanent molds having mold cavities measuring 3cm in height with each mold cavity having a top diameter of 5.5cm and a bottom diameter of 5cm.

Alloys AC2, AC4, AC6, AC9 and AC10

Five different alloys were prepared in accordance with the test procedure detailed above for Alloys AD9 - AD14.

Chemical analysis samples were taken from the melt and analyzed using ICP mass spectrometry. The chemical composition of the prepared alloys, namely - AC2, AC4, AC6, AC9 and AC10, are shown in Table 1 hereinbelow. The recovery rate of strontium was determined to be 87-92%.

The molten metal was permanent mold cast using an H-13 (mild) steel permanent mold. The mold contained cavities for two ASTM standard test bars each measuring 14.2cm in length and 0.7cm in depth or thickness. Grip width was 1.9cm while gage length and gage width was 5.08cm and 1.27cm, respectively. The mold was provided with a sprue, riser and gating system to bottom-feed the two tensile bar cavities.

15

10

TABLE 1

ALLOY	CHEM	ICAL CO	OMPOSI	TION					
	Al, wt%	Sr, wt%	Mn, wt%	Zn (ppm)	Fe (ppm)	Cu(ppm)	Ni (ppm)	Si (ppm)	Ca (ppm)
AM50	5.0	-	0.32	200	20	10	10	70	20
90-10 Sr-Al aster alloy	10	90							
A1 ·	4.90	1.74	0.26	94	23	4	. 3	34	18
A2	4.85	1.23	0.29	94	11	2	3	47	17
AD9	4.96	0.94	0.28	56	<10	<2	<2	-	. 17
AD10	5.07	1.21	0.29	61	₹10	<2	<2		18,
AD11	5.00	1.54	0.28	54	<10	<2	<2	-	18
AD12	5.18	2.31	0.28	54	<10	<2	<2	<u> </u>	18

ALLOY	СНЕМ	CAL CO	OMPOSI	TION					
AD13	5.10	3.77	0.28	54	< 10	<2	< 2	-	18
AD14	5.71	6.89	0.28	54	< 10	<2	<2	• .	18
AC2	4.90	1.59	0.30	43	60	<2	<2	•	< 10
AC4	4.70	1.26	0.33	78	84	122	7 .	•	35
AC6	4.89	1.22	0.32	69	41	127	9	-	40
AC9	4.82	1.07	0.32	42	39	82	3	•	31
AC10	5.08	1.46	0.29	52	39	150	2	-	8

Various properties of the alloys were then tested as set forth below and compared against other magnesium alloys and aluminum alloy A380.

Test Methods

The diecast and permanent mold cast test specimens were subjected to the following tests:

10

5

Creep Resistance or Creep Extension

The creep resistance of the diecast and permanent mold cast test specimens was measured in accordance with ASTM E139-83. In particular, test specimens were exposed to air for a period of 60 minutes and then subjected, for a period of 200hr, to a constant stress of 35 MPa via an Applied Test Systems, Inc. (ATS) Lever Arm Tester-2320 creep testing machine while being maintained at a temperature of 150°C. The gage length of each test specimen was then measured and the difference between the original gage length (i.e., 1.27cm) and the gage length of each specimen at the end of the 200hr test period was determined. The difference in gage

20

25

length determined for each test specimen was then divided by 1.27cm and the result reported as a percent (%).

Bolt-Load-Retention or Bolt-Load-Loss

The bolt-load-retention of the diecast test specimens was measured in accordance with the following procedure: diecast cylinders of the alloys were used to machine disc samples measuring 25.4x9mm. A hole having a diameter of 8.4mm was then drilled in the middle of each sample. An M8 steel bolt and nut (1.25 pitch) were then screwed with a torquewrench into each disc sample using a washer of 15.75mm OD and 8.55 ID and torqued to 265 lbs.in (30Nm). A special set-up was used to measure the initial angle to which the bolt had to be rotated to reach the prescribed torque.

The special set-up consisted of a 360° mild steel protractor fabricated by the machine shop at Noranda Inc. Technology Center. The protractor had a central hole in the shape of an M10 nut, machined to receive and fix the test specimen in place. A machined M8 socket was used to adapt the hole to an M8 bolt. The protractor was bolted to a table to counteract the rotation force applied during torquing with a digital torque wrench (model Computorq II -64-566 manufactured by Armstrong Tool, USA).

The bolted samples were then immersed in an oil bath having a temperature of 150°C and were kept in the oil bath for 48 hours where the bolts lost some torque due to stress relaxation. The samples were then removed from the oil bath, cooled to room temperature and the bolts retightened to the initial torque of 265 lbs.in (30Nm). The additional angle required to reach the initial torque was then measured and this value used as a measure of bolt-loosening. The results are reported in degrees (°).

The bolt-load-retention of the permanent mold cast test specimens was measured in accordance with the following procedure:

10

20

permanent mold cast disc samples of the alloys were machined to discs measuring 35x11mm. A hole having a diameter of 10.25 was then drilled in the middle of each sample. An M10 steel bolt and nut (1.5 pitch) were then screwed with a torque-wrench into each disc sample using a washer of 19.75mm OD and 10.75 ID and torqued to 440 lbs.in (50Nm). A special setup was used to measure the initial angle to which the bolt had to be rotated to reach the prescribed torque. The set-up was identical to that noted above, except that a machined M8 bolt was not used to adapt the central hole to the M8 bolt. The bolted samples were then immersed in an oil bath having a temperature of 150°C and were kept in the oil bath for 48 hours where the bolts lost some torque due to stress relaxation. The samples were then removed from the oil bath, cooled to room temperature and the bolts retightened to the initial torque of 440 lbs.in (50Nm). The additional angle required to reach the initial torque was then measured and this value used as a measure of bolt-loosening. The results are reported in degrees (°).

Tensile Properties

Tensile properties (i.e., tensile yield strength, ultimate tensile strength and elongation) at an elevated temperature of 150°C and at room temperature were measured in accordance with ASTM E8-99 and E21-92. An Instron servovalve hydraulic Universal Testing Machine (model number 8502-1988) equipped with an Instron oven (model number 3116) and an Instron extensiometer (model number 2630-052) were used in conjunction with the subject test methods.

For tensile testing at 150°C, test specimens were clamped within the test assembly and heated to a temperature of 150°C and then maintained at this temperature for a period of 30 minutes. Specimens were then tested at 0.13cm/cm/min through yield and at 1.9cm/min to failure.

15

20

25

For room temperature tensile testing, specimens were tested at 0.7MPa/min through yield and at 1.9cm/min to failure.

Tensile yield strength was determined by passing a tangent to the part of the stress-strain curve between 20.5-34.5 MPa and by passing a second line parallel to the one intersecting the y-axis at a 0.2% extension. Results are reported in megapascals (MPa).

Ultimate tensile strength was determined as the stress at rupture or as the maximum stress in the stress-strain curve. Results are reported in MPa.

Elongation was determined by measuring the gage length of each test specimen before and after testing. Results are reported in percent (%).

Salt-Spray Corrosion Resistance

The resistance of the diecast corrosion test plate test specimens to corrosion was measured in accordance with ASTM B117. In particular, specimens were cleaned using a 4% NaOH solution at 80°C, rinsed in cold water and dried with acetone. The specimens were then weighed and then vertically mounted at 20° from the vertical axis within a SINGLETON salt-spray test cabinet (model number SCCH #22). The vertically mounted specimens were then exposed to a 5% NaOH/distilled water fog for a period of 200hr. During the test period, the fog tower was adjusted to a collection rate of 1cc/hr and the parameters of the cabinet checked every 2 days. At the end of the 200hr test period, the specimens were removed, washed in cold water and cleaned in a chromic acid solution (i.e., chromic acid containing silver nitrate and barium nitrate) as per ASTM B117. The samples were then re-weighed and the weight change per sample determined. The results are reported in milligrams per square centimeter per day (mg/cm²/day).

EXAMPLES 1 AND 2 AND COMPARATIVE EXAMPLES C1 TO C5

In these examples diecast specimens prepared in accordance with the teachings of the present invention and diecast magnesium alloys AZ91D, AE42, AS41 and AM60B and aluminum alloy A380 were tested for creep resistance, bolt-load retention, various tensile properties at both room temperature and at 150°C and salt-spray corrosion resistance. The results are tabulated in Table 2.

TABLE 2

Summary of Examples 1 and 2 and Comparative Examples C1 to C5

	minary of	·			·		
EXAMPLE	1	2	C1	C2	СЗ	C4	C5
ALLOY	A1	A2 -	AZ91D	AE42	AS41	AM60B	A380
Properties:	· .						<u> </u>
Creep Extens	ion (%) at	150°C					
Run 1	0.05%	0.12%	1.64%	0.09%	0.168 %	-	0.192%
Run 2	0.03%	0.07%	0.90%	0.06%	0.102 %	-	0.154%
Run 3	0.02%	0.02%	1.08%	0.05%	0.12%	<u>-</u>	0.18%
AVERAGE	0.03%	0.06%	1.21%	0.07%	0.13%	<u>.</u>	0.18%
Bolt-Load-Los	ss (°) at 150	°C					
Run 1	6.0°	6.0°	1,4.0°	9.0°	10.5°	· -	2.0°
Run 2	6.0°	6.5°	14.5%	7.5°	11.0°	•	2.0°
AVERAGE	6.0°	6.3°	14.3°	8.3°	10.8°		2.00

*							
EXAMPLE	1	2	C1	C2	C3	C4	C5
Tensile Proper	ties at 150	i. C	A291	AE42	2541	AM60	D3.80
Yield Strength	(MPa)	,				· ·	·
Run 1	119.9	100.8	108.2	85.4	87.7		168.5
Run 2	111.1	105.0	99.5	96.2	96.3	·	147.6
Run 3	112.8	100.0	104.4	87.2	92.0		152.0
Run 4	108.5	106.0	-	85.0	98.4		146.5
Run 5	106.9	100.0	106.9	89.7	89.6		158.6
Run 6	100.0	96.6	106.9	82.8	89.6	·	148.2
Run 7	103.4	96.6	103.4	86.2	93.1		137.9
AVERAGE	108.9	100.7	104.9	87.5	92.4		151.3
Ultimate Tens	ile Strengt	h (MPa)		•		,	
Run 1	188.3	150.8	179.9	139.0	154.0		293.0
Run 2	168.1	143.3	161.6	162.6	153.0		235.7
Run 3	171.1	149.7	174.3	152.3	155.3		264.3
Run 4	161.1	157.9	-	143.5	147.9		259.9
Run 5	158.6	148.3	169.0	137.9	144.8		251.7
Run 6	158.6	144.8	169.0	127.6	137.9		255.1
Run 7	151.7	- 148.3	165.5	137.9	155.1		220.6
AVERAGE	165.4	149.0	169.9	143.0	149.7		254.3
Elongation %							
Run 1	11.7	19.3	20.6	16.1	19.8		4.4
Run 1	11.7	19.3	20.6	16.1	19.8		4.4

EXAMPLE	1	2	C1	C2	C3	C4	C5.
Ru 2	8.0	9.2	12.5	24.4	20.4		3.1
Ru 3	22.0	17,6	12.6	30.2	19.5		7.5
Rn 4	8.2	24.9		25.6	7.4		7.5
Run5	22.1	11.7	19.5	21.6	17.6	·	4.5
Rn 6	14.3	23.4	11.7	22.3	16.7		7.9
Run7	7.8	19.5	19.5	24.6	17.8		4.5
AERAGE	13.4%	17.9%	16%	23.5%	17%	·	6.7%
Tnsile Propert	ies at Roo	m Temperat	ure				
Yiel Strength (MPa)						
1	136.7	136.6	154.1	132.0	118.1		141.9
Ru 2	146.0	136.2	156.9	131.5	139.3		157.8
Run	139.7	136.2	150.8	130.9	136.8		160.6
Rn 4	146.6	136.0	154.8	131.2	135. <u>7</u>		156.4
Run 5	136.2	135.3	-	131.0	129.6		155.9
Run 6	151.7	141.4	162.1	137.9	148.2		162.0
Run 7	144.8	137.9	158.6	137.9	151.7		148.2
Run 8	148.3	141.4	158.6	137.9	131.0		158.6
AVERAGE	143.7	137.6	1,56.6	133.8	123.8		155.2
Ultimate Tens	ile Strengt	h (MPa)					
Run 1	206.8	228.0	257.0	240.3	255.4		247.4
Run 2	215.5	223.1	249.4	221.6	231.0	·	233.0
		L					*

•							
EXAMPLE	1	2	,CI	C2 AF	C3 A S	C4 DM(i)	C5
Run 3	215.3	236.5	220.7	212.8	241.5		332.5
Run 4	222.9	228.5	231.5	240.3	254.6		312.1
Run 5	241.6	238.2	-	240.7	262.6	ζ·	323.5
Run 6	186.2	231.0	231.0	206.9	196.5		310.3
Run 7	-	234.5	227.6	227.6	217.2		251.7
Run 8	193.1	241.4	248.3	224.1	231.0		317.2
AVERAGE	211.6	232.7	237.9	226.8	236.3		291.0
Elongation %							
Run 1	3.7	7.6	5.6	13.2	11.0		1.8
Run 2	4.1	6.4	4.4	8.3	5.4		1.7
Run 3	5.0	9.2	3.6	5.6	8.0	·	4.7
Run 4	5.0	8.2	3.5	12.4	9.8		4.0
Run 5	7.9	8.4	4.3	10.2	10.1		3.0
Run 6	3.7	6.2	5.0	6.2	3.3		4.4
Run 7	2.5	11.2	5.0	10.0	4.4		2.2
Run 8	2.5	11.2	6.2	8.7	7.8		3.4
AVERAGE	4.3%	8.6%	4.7%	9.3%	7.4%		3.2%
Salt-Spray C	orrosion Ra	ate (mg/cm²	/day)	•			
Run 1	0.104	0.119	0.127	0.172	0.019	0.307	0.322
Run 2	0.097	0.105	0.097	0.251	0.174	0.236	0.330
Run 3	0.057	0.197	0.085	0.144	0.317	0.175	0.380
1		l					

10

15

20

			A2 19	AE	42	DOMA	A380
EXAMPLE	1	2	C1	C2	C3	C4	C5
AVERAGE	0.086	0.155	0.103	0.189	0.170	0.260	0.344

A review of the average creep extension, bolt-load-loss, tensile properties and salt-spray corrosion rate values in Table 2 indicates that the magnesium-based casting alloys of the present invention have improved overall elevated temperature performance as compared to magnesium alloys AZ91D, AE42, AS41 and AM60B and aluminum alloy A380.

In particular, Examples 1 and 2 demonstrated improved creep resistance over comparative Examples C1(AZ91D), C2(AE42) and C5(A380) and better bolt-load retention (smaller angle of loss) than Comparative Examples C1 to C3(AZ91D, AE42 and AS41).

In terms of tensile properties, Examples 1 and 2 demonstrated improved yield strength (at room temperature and at 150°C) over Comparative Examples C2(AE42) and C3(AS41) and improved elongation (at room temperature and at 150°C) over Comparative Example C5(A380).

Examples 1 and 2 further demonstrated improved salt-spray corrosion resistance over Comparative Examples C2(AE42), C3(AS41), C4(AM60B) and C5(A380) and comparable salt-spray corrosion resistance to that demonstrated by Comparative Example C1(AZ91D).

EXAMPLES 3 TO 8 AND COMPARATIVE EXAMPLES C6 TO C10

In these examples permanent mold cast disc specimens prepared in accordance with the present invention and permanent mold cast magnesium alloys AZ91D, AM50, AS41 and AE42 and aluminum alloy A380 were tested for bolt-load retention. The results are tabulated in Table 3.

TABLE 3
Summary of Examples 3 to 8 and Comparative Examples C6 to C10

EXAMPLE	3	4	5 .	6	7	8	C6	C 7	C8	Cá	C10
ALLOY	AD90	AD10	AD11	AD12	AD13	AD14	AZ91D	AM50	AS41	AE42	A380
Properties											
Bolt-Load-l	.oss (°)										
Run 1	3.25°	2.5°	2.5° -	4.5°	2.0°	2.0°	9.5°	4.75°	3.0°	3.0°	2.0°
Run 2	2.75°	3.0°	3.0°	3.0°	2.5°	2.0°	9.5°	7.5°	6.0°	3.0°.	2.0°
Run 3	-	-	. -	-	-	-	8.5°	7.0°	-	4.5°	-
Run 4	. -	-			-	-	9.5°	7.5°	-	3.5°	-
Run 5	-	-	-	-	-	-	8.5°	-		7.0°	-
AVERAGE	3.0°	2.75°	2.75°	3.75°	2.25°	2.0°	9.1°	6.7°	4.5°	4.2°	2.0°

By way of the average bolt-load-loss values shown in Table 3, it can be seen that the permanent mold cast alloys of the present invention (i.e., Examples 3 to 8) demonstrate improved bolt-load retention (smaller angle of loss) when compared to magnesium alloys AZ91D, AM50, AS41 and AE42 (i.e., C6 to C9) and comparable bolt-load retention to that demonstrated by aluminum alloy A380 (i.e., C10).

EXAMPLES 9 TO 12 AND COMPARATIVE EXAMPLES C11 TO C13

. 10

15

In these examples permanent mold cast ASTM standard flat tensile specimens prepared in accordance with the present invention and permanent mold cast magnesium alloys AZ91D and AE42 and aluminum alloy A380 were tested for creep resistance. The results are tabulated in Table 4.

TABLE 4
Summary of Examples 9 to 12 and Comparative Examples C11 to C13

Run 1	0.012%	0.000 %	0.0213 %	0.03 %	0.130 %	0.035%	0.092%
	0.000	0.006%	0.0215%	0.03%	0.136%	0.035%	
Creep Exten	sion (%) at 1	50°C					
Properties:							
ALLOY	AC9	AC4	AC6	AC10	AZ91D	AE42	A380
EXAMPLE	9	10	11	12	C11	C12	C13

By way of the average creep extension values shown in Table 4, it can be seen that the permanent mold cast alloys of the present invention (i.e., Examples 9 to 12) demonstrate improved creep resistance at 150°C when compared to magnesium alloys AZ91D and A380 (i.e., C11 and C13) and comparable creep resistance to that demonstrated by magnesium alloy AE42 (i.e., C12).

EXAMPLES 13 TO 16 AND COMPARATIVE EXAMPLES C14 TO C16

In these examples permanent mold cast ASTM standard flat tensile specimens prepared in accordance with the present invention and permanent mold cast magnesium alloys AZ91D and AE42 and aluminum alloy A380 were tested for tensile properties at 150°C. The results are tabulated in Table 5.

TABLE 5
Summary of Examples 13 to 16 and Comparative Examples C14 to C16

XAMPLE	13	14	15	16	C14	C15	C16
ILLOY	AC9	AC6-AC4	AC10	AC2	AZ91D	AE42	A380
Properties:		<u> </u>					
Tensile Properties at	150°C						
rield Strength (MPa)						
Run 1	56.5	59.3	62.0	69.7	81.2	43.9	124.3
Run 2	58.6	66.7	62.1	62.9	78.7	48.0	126.4
Run 3	-	66.5		-	79.4	43.4	•
Run 4	-	-		-	93.1	44.8	-
AVERAGE	57.6	64.2	62.1	66.3	83.1	45.0	125.4
Ultimate Tensile St	rength (MP	a)					
Run 1	118.0	96.4	100.0	95.5	169.9	111.0	187.5
Run 2	-	95.5	117.2	99.9	176.7	113.2	162.4
Run 3	-	89.7		-	166.5	113.4	-
Run 4			-		162.1	117.2	-
AVERAGE	118.0	93.9	108.6	97.70	168.8	113.6	175.0
Elongation %		<u></u>					
Run 1	5.7	4.6	3.1	1.9	5.6	10.5	1.3
Run 2	-	-	5.5	2.6	11.0	11.3	0.9
Run 3	-	2.5	-	-	8.7	11.0	-
		 			1		

AVERAGE	5.7%	3.6%	4.3%	2.3%	8.6%	9.0%	1.1%
Run 4	-	•	. :	•	9.0	3.0	<u>.</u>
EXAMPLE	13	14	15	16	C14	C15	. C16

By way of the average tensile properties values shown in Table 5, it can be seen that the permanent mold cast alloys of the present invention (i.e., Examples 13 to 16) demonstrate improved yield strength at 150°C when compared to magnesium alloy AE42 (i.e., C15).

Having thus described the invention, what is claimed is:

CLAIMS

5

10

- 1. A magnesium-based casting alloy having improved elevated temperature performance which comprises, in weight percent, 2 to 9% aluminum and 0.5 to 7% strontium with the balance being magnesium except for impurities commonly found in magnesium alloys.
- 2. The magnesium-based casting alloy of Claim 1, wherein said alloy comprises 4 to 6% aluminum.
- 3. The magnesium-based casting alloy of Claim 1, wherein said alloy comprises 4.5 to 5.5% aluminum.
- 4. The magnesium-based casting alloy of Claim 1, wherein said alloy comprises 1 to 5% strontium.
- The magnesium-based casting alloy of Claim 1, wherein said alloy comprises 1 to 3% strontium.
 - 6. The magnesium-based casting alloy of Claim 1, wherein said alloy

comprises 1.2 to 2.2% strontium.

- 7. The magnesium-based casting alloy of Claim 1, wherein said alloy has a structure including a matrix of grains of magnesium having a mean particle size of from about 10 to about 200μm reinforced by intermetallic compounds having a mean particle size of from about 2 to about 100μm.
- 8. The magnesium-based casting alloy of Claim 1, wherein said alloy is a diecast alloy having an average % creep deformation at 150°C of less than or equal to 0.06%, an average bolt-load-loss at 150°C of less than or equal to 6.3°, and an average tensile yield strength at 150°C of greater than 100MPa.

10

15

- 9. The magnesium-based casting alloy of Claim 1, wherein said alloy is a permanent-mold cast alloy having an average % creep deformation at 150°C of less than or equal to 0.03%, an average bolt-load-loss at 150°C of less than or equal to 3.75°, and an average tensile yield strength at 150°C of greater than 57MPa.
- 10. A magnesium-based casting alloy having improved elevated temperature performance which comprises, in weight percent, 2 to 9% aluminum, 0.5 to 7% strontium, 0 to 0.60% manganese, and 0 to 0.35% zinc, with the balance being magnesium except for impurities commonly found in magnesium alloys.
- 11. The magnesium-based casting alloy of Claim 10, wherein said alloy comprises 4 to 6% aluminum.
- 12. The magnesium-based casting alloy of Claim 10, wherein said alloy comprises 4.5 to 5.5% aluminum.
- 13. The magnesium-based casting alloy of Claim 10, where said alloy comprises 1 to 5% strontium.
- 14. The magnesium-based casting alloy of Claim 10, where said alloy comprises 1 to 3% strontium.
- 15. The magnesium-based casting alloy of Claim 10, where said alloy comprises 1.2 to 2.2% strontium.
 - 16. The magnesium-based casting alloy of Claim 10, wherein said alloy comprises 0.25 to 0.35% manganese.
 - 17. The magnesium-based casting alloy of Claim 10, wherein said alloy comprises 0.28 to 0.35% manganese.
- 25 18. The magnesium-based casting alloy of Claim, 10, wherein said alloy comprises 0 to 0.1% zinc.
 - 19. The magnesium-based casting alloy of Claim 10, wherein said alloy comprises 0 to 0.05% zinc.

15

20

25

- 20. The magnesium-based casting alloy of Claim 10, wherein said alloy has a structure including a matrix of grains of magnesium having a mean particle size of from about 10 to about 200 μ m reinforced by intermetallic compounds having a mean particle size of from about 2 to about 100 μ m.
- 21. The magnesium-based casting alloy of Claim 10, wherein said alloy is a diecast alloy having an average % creep deformation at 150°C of less than or equal to 0.06%, an average bolt-load-loss at 150°C of less than or equal to 6.3°, and an average tensile yield strength at 150°C of greater than 100MPa.
- 22. The magnesium-based casting alloy of Claim 10, wherein said alloy is a permanent-mold cast alloy having an average % creep deformation at 150°C of less than or equal to 0.03%, an average bolt-load-loss at 150°C of less than or equal to 3.75°, and an average tensile yield strength at 150°C of greater than 57MPa.
- 23. A magnesium-based casting alloy comprising, in weight percent, 4 to 6% aluminum, 1 to 5% strontium, 0.25 to 0.35% manganese, and 0 to 0.1% zinc with the balance being magnesium except for impurities commonly found in magnesium alloys.
- 24. The magnesium-based casting alloy of Claim 23, wherein said alloy has a structure including a matrix of grains of magnesium having a mean particle size of from about 10 to about 200 μ m reinforced by intermetallic compounds having a mean particle size of from about 2 to about 100 μ m.
- 25. The magnesium-based casting alloy of Claim 23, wherein said alloy is a diecast alloy having an average % creep deformation at 150°C of less than or equal to 0.06%, an average bolt-load-loss at 150°C

15

of less than or equal to 6.3°, and an average tensile yield strength at 150°C of greater than 100MPa.

- 26. The magnesium-based casting alloy of Claim 23, wherein said alloy is a permanent-mold cast alloy having an average % creep deformation at 150°C of less than or equal to 0.03%, an average bolt-load-loss at 150°C of less than or equal to 3.75°, and an average tensile yield strength at 150°C of greater than 57MPa.
- 27. A magnesium-based casting alloy comprising, in weight percent, 4 to 6% aluminum, 1 to 3% strontium, 0.25 to 0.35% manganese, and 0 to 0.1% zinc with the balance being magnesium except for impurities commonly found in magnesium alloys.
- 28. A magnesium-based casting alloy comprising, in weight percent, 4.5 to 5.5% aluminum, 1.2 to 2.2% strontium, 0.28 to 0.35% manganese, and 0 to 0.05% zinc with the balance being magnesium except for impurities commonly found in magnesium alloys.

INFORMATION ON PAIGHT IZMBY MEMBERS			
	INI OFFICE OF	Dalent Iamur	mempers

PCT/CA 00/01527

Patent document cited in search report		Publication date	.1	Patent family m mber(s)	Publication date
US 5340416	A	23-08-1994	JP JP DE DE EP	3110117 B 5171331 A 69221712 D 69221712 T 0549998 A	20-11-2000 09-07-1993 25-09-1997 12-02-1998 07-07-1993
EP 0665299	A	02-08-1995	JP DE DE US	7224344 A 69423335 D 69423335 T 6143097 A	22-08-1995 13-04-2000 30-11-2000 07-11-2000

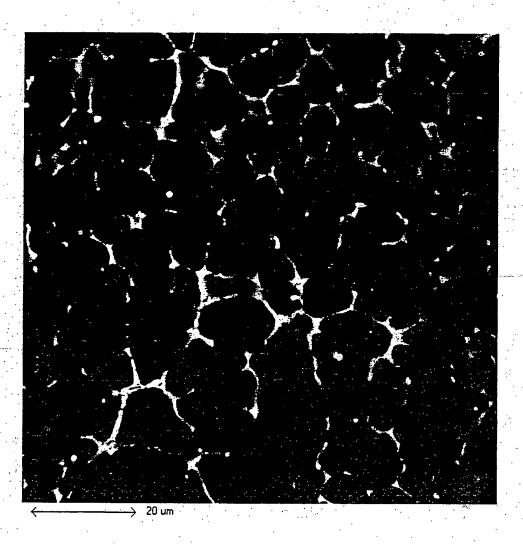


FIG. 1

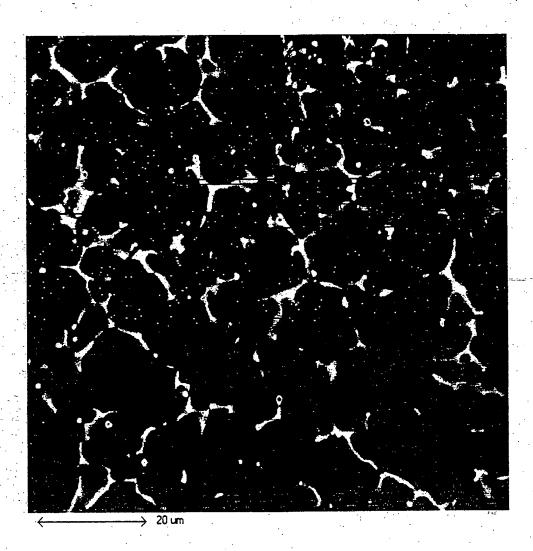


FIG. 2

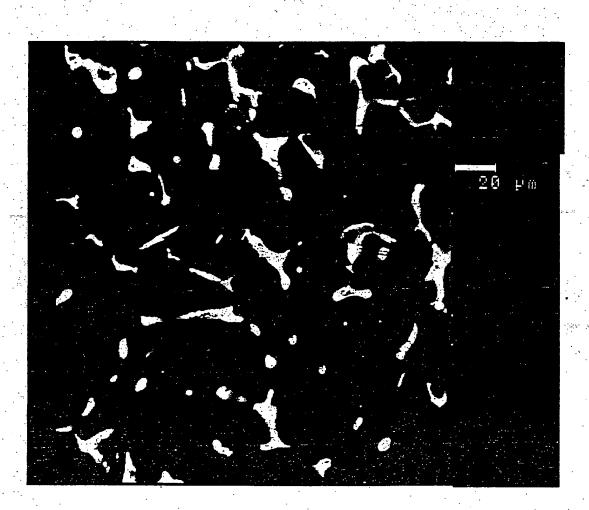


FIG. 3

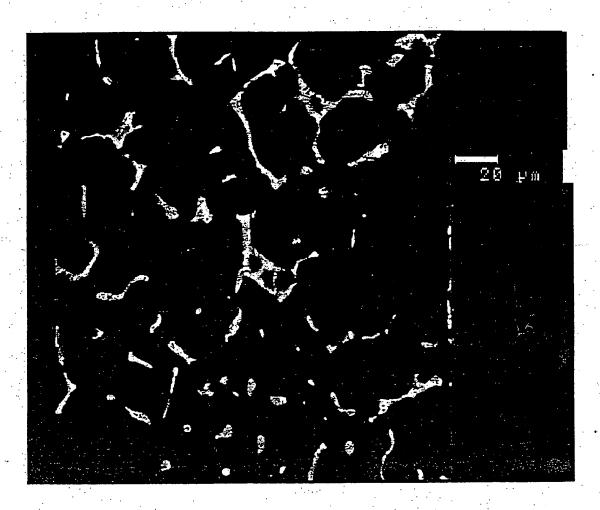


FIG. 4